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METHODS FOR THE DETERMINATION OF WATER IN PETROLEUM AND ITS PRODUCTS

BY

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METHODS FOR THE DETERMINATION OF WATER IN PETROLEUM AND ITS PRODUCTS.

By Irving C. Allen and Walter A. Jacobs.

INTRODUCTION.

This paper is issued by the Bureau of Mines in response to numerous inquiries regarding methods for the determination of water in fuel oils and in petroleum products, and is part of a report on petroleum technology. This report, issued chapter by chapter, aims to describe how petroleum products can be improved in quality, how they can be more efficiently utilized, and how some materials now regarded as waste products can be made useful.

THE WATER CONTENT OF OIL.

Water in a lubricating oil sometimes forms emulsions that tend to lower the durability of the oil and make it less efficient as a lubricant. In fuel oils the unfavorable effect of water is even greater. A fuel oil containing considerable water when burned under a steam boiler gives an unsteady flame that tends to go out. Water, being noncombustible, reduces the heating value of an oil in direct ratio to the percentage present, and converted into steam increases the inert and valueless gases within the firebox, reducing the heating effect of the combustible gases that should normally occupy the firebox space.

A fuel oil containing water when burned in an internal-combustion engine will be even more unsatisfactory. A drop of water may prevent ignition of the fuel, and a number of such failures of the fuel to ignite would stop the engine.

The importance therefore of accurately determining the water content of an oil before use is recognized. Various methods for the determination of water are therefore described in the following pages.

CLASSIFICATION OF METHODS FOR THE DETERMINATION OF WATER.

Methods employed for the determination of water in petroleum and its products may be classified as follows:

1. Ascertaining the loss of weight by heating.

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2. Diluting with a solvent and separating by gravity.
3. Diluting with a solvent and separating with a centrifuge.
4. Treating with calcium carbide.
5. Treating with sodium.
6. Use of a color-comparator tube.
7. Treating with normal acids.
8. Electrical treatment.
9. Distilling with a nonmiscible liquid.
10. Directly distilling off the water.

In this report the above-mentioned methods for the determination of water in petroleum and in its products are taken up in order, a discussion of each, and conclusions as to its value are given, and a bibliography bearing on the general subject is presented.

1. ASCERTAINING THE LOSS OF WEIGHT BY HEATING.

Conradson [a] determines the water content of heavy oils by the loss in weight on heating. However, he recognizes the fact that whenever a heavy oil is heated to a temperature of 100° C. some oil is volatilized and lost.

Wright [b] heats to from 105° C., Hurst [c] to 220° F., and Archbutt and Deeley [d] to from 105° to 110° C.; Davis [e] saturates a tared filter paper and heats to 110° C.; Patrick [f] heats the oil until slight decomposition takes place and assumes the loss in weight to be a loss of water.

Gavalovski [g] has improved the evaporation method by using duplicate samples in tared flasks. He leaves one flask open in the usual manner and attaches to the other a sulphuric-acid drying tube, which, he states, absorbs the water and allows the oil vapors to pass through unabsorbed. The difference in loss of weight on heating, he assumes to be a water loss. Lowenstein [h] dilutes with 95 per cent alcohol and dries repeatedly on a water bath and then at a temperature of 105° C. to constant weight.

---

[a] Conradson, F. P., Determination of water in greases: Jour. Am. Chem. Soc., vol. 26, June, 1904, p. 705; Jour. Ind. and Eng. Chem., vol. 1, June, 1909, p. 355.

[b] Wright, A. C., Analysis of oils and allied substances, 1903, p. 104. Jour. Ind. and Eng. Chem., vol. 1, June, 1909, p. 355.

[c] Hurst, G. H., Determination of water in greases: Jour. Ind. and Eng. Chem., vol. 1, June, 1909, p. 355.

[d] Archbutt, L., and Deeley, R. M., Lubrication and lubricants, 1900, pp. 293–294: Jour. Ind. and Eng. Chem., vol. 1, June, 1909, p. 355.

[e] Davis, C. B., Elimination and determination of water in oils, fats, and waxes: Jour. Am. Chem. Soc., vol. 23, 1901, pp. 487–488; Jour. Soc. Chem. Ind., vol. 20, September 30, 1901, p. 941.

[f] Patrick, G. E., Apparatus for rapid determination of water in butter. Jour. Am. Chem. Soc., vol. 28, 1906, pp. 1611–1616; Chem. Zeit., vol. 32, 1908, p. 1176; Chem. Abs., vol. 1, 1907, p. 70, vol. 3, 1909, p. 504.

[g] Gavalovski, A., Crude oil and water: Österr. chem. tech. Zeit., vol. 27, p. 3; Chem. Abs., vol. 3, May 10, 1909, p. 1078.

[h] Lowenstein, A., The rapid determination of moisture in commercial products of a viscous or semisolid consistency. Jour. Ind. and Eng. Chem., vol. 1, 1909, pp. 252–253; Chem. Abs., vol. 3, Sept. 10, 1909, p. 1971.

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The evaporation method, varied as described above, is approximate and applicable only to heavy oils and greases. Its accuracy even with heavy greases is questionable.

2. DILUTING WITH A SOLVENT AND SEPARATING BY GRAVITY.

Curtis and Thompkins [a] dilute 200 c. c. of the oil with 600 c. c. of gasoline (specific gravity, 0.7000), allow the liquid to settle in a graduated glass tube, and read the volume of water settling to the bottom of the tube. Sussanow [b] in using this method employs benzene instead of gasoline as a solvent.

This method is only approximate. Its accuracy is modified by the quantity and quality of the diluent used, by the amount of mineral matter in the oil, by the character of the insoluble portions of the oil itself, such as asphaltine, and by the length of time allowed for settling.

3. DILUTING WITH A SOLVENT AND SEPARATING WITH A CENTRIFUGE.

Charitschkoff,[c] Wieleczynski,[d] Rosenthal,[e] and O'Neill [f] have greatly improved the method described under 2. After diluting the oil with an equal volume of benzene they whirl it vigorously until the separated layer of water does not appear to increase in volume. However, as water is somewhat soluble in any diluent used and also in oils, a portion of the water content will fail to appear, consequently the method in which a diluent is used can not be considered accurate. It is advisable first to agitate the diluent vigorously with water and then to separate with the centrifuge in order to saturate it with water before using.

Groschuff [g] states that 100 grams of benzene will dissolve 0.03 gram of water at 3° C. and 0.337 gram of water at 77° C., whereas petroleum products (density 0.792) will dissolve from 0.0012 gram at 2° C. to 0.097 gram at 94° C.

---

[a] Curtis, M., and Thompkins, P. W., Notes on the determination of water in California crude oils: Jour. Soc. Chem. Ind., vol. 21, Dec. 31, 1902, p. 1519.

[b] Sussanow, J., Über die Defekte der Benzinmethode der Wasserbestimmung im Erdöl: Neft. Djelo., No. 8, 1910, pp. 7–10.

[c] Charitschkoff, K. W., Bestimmung von suspendierten Wasser in dem Naphtha: Chem. Zeit., vol. 30, 1906, p. 93; Westnik Shiroff Weschtnich, vol. 1, 1905, p. 187.

[d] Wieleczynski, M., Wasserbestimmung in Rohöl: Petroleum, vol. 3, 1907, p. 285.

[e] Rosenthal, von, Über die Wasser- und Schmandbestimmung in Erdölen. Chem. Zeit., vol. 33, 1909, p. 1259; Petroleum, vol. 6, 1909, p. 22.

[f] O'Neill, Edmund, The determination of water and asphaltine in petroleum: Cal. Derrick, vol. 3, June, 1910, p. 3; December, 1910, p. 5; Petroleum, vol. 6, April, 1911, pp. 777–778; vol. 5, August, 1910, p. 1393.

[g] Groschuff, E., The solubility of water in benzene, petroleum, and paraffin oil: Chem. Abs., vol. 5, Aug. 10, 1911, p. 2550.

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4. TREATING WITH CALCIUM CARBIDE.

Dupré,[a] Danne,[b] O. Masson,[c] and I. Masson [d] agitate samples of the various materials containing water under examination with an excess of calcium carbide, measure the volume of acetylene evolved, and calculate the content of water.

Wieleczynski[e] and Roberts and Fraser[f] maintain that this method may be applied successfully to petroleum. The method may be carried out conveniently as follows:

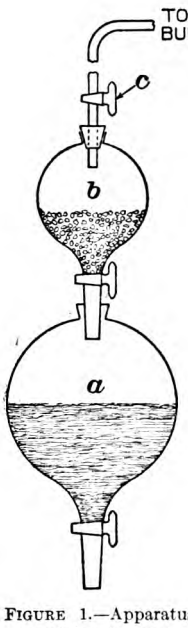

FIGURE 1.—Apparatus for determining the water content of petroleum and its products by treating with calcium carbide.

To the stopper of a 250-c. c. separatory funnel such as *a*, figure 1, is fitted a second 100-c. c. separatory funnel *b*, which in turn is fitted with a small glass stopcock *c*.

17.24 grams of the sample is run into *a*, the cock of *b* is closed, and *b* is connected to *a*. About 10 grams of powdered calcium carbide is run into *b* and cock *c* inserted. The cock of *b* is cautiously opened, the dropping of any carbide into *a* being avoided, and the whole is immersed in a water bath maintained at any convenient temperature *t*. Immersion is made up to cock *c*, which is open to the atmosphere. When the temperature *t* has been reached, *c* is closed, the apparatus is removed from the bath, and the carbide is shaken into *a*. The sample and carbide are then shaken vigorously and, if necessary, are cooled several times under cold water. After the reaction is completed, the whole is again brought to temperature *t* by immersion in the water bath, connection is made between *c* and a gas burette, and the increase in volume due to the generation of acetylene is measured. One gram of water generates approximately 580 c. c. of acetylene [g] at 0° C. and 760 mm. pressure.

---

[a] Dupré, P. V., Determination of water in cordite by means of calcium carbide. Analyst, vol. 31, 1906, p. 213.

[b] Danne, H. A., The estimation of water in organic matter by means of calcium carbide: Address before the Society of Chemical Industry of Victoria in 1911.

[c] Masson, O., Determination of moisture in sheep's wool by action of calcium carbide. Proc. Soc. Chem. Ind. Victoria, 1909.

[d] Masson, I., The use of calcium carbide for determining moisture. Chem. News, vol. 103, 1911, pp. 37–38; Chem. Abs., vol. 5, Apr. 20, 1911, p. 1377; Determination of water of crystallization by action of calcium carbide. Trans. Chem. Soc., vol. 97, 1910, pp. 851–867.

[e] Wieleczynski, M., Determining impurities (water) in Boryslaw (Galicia) petroleum. Naphtha, vol. 12, 1904, pp. 23–25; Jour. Soc. Chem. Ind., vol. 23, May 16, 1904, p. 504.

[f] Roberts, R. W., and Fraser A., Easy process for estimating water in petroleum. Jour. Soc. Chem. Ind., vol. 29, 1910, pp. 197–198; Chem. Abs., vol. 4, June 20, 1910, p. 1664; Petroleum, vol. 5, July 6, 1910, p. 1173; vol. 6, Sept. 6, 1911, p. 2245; Matirières Grasses, 1911, p. 4; Met. and Chem. Eng., vol. 8, May, 1910, p. 283.

[g] Masson, I., Determination of water of crystallization by action of calcium carbide: Trans. Chem. Soc., vol. 97, 1910, pp. 851–867: The use of calcium carbide for determining moisture: Chem. News, vol. 103, pp. 37–38; Chem. Abs., vol. 5, Apr. 20, 1911, p. 1377; Roberts., R. W., and Fraser, A., Easy process for

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It should be noted that acetylene is soluble in oils. One c. c. of kerosene (specific gravity 0.7950) will dissolve approximately 1.375 c. c. of acetylene, and 1 c. c. of spindle oil of specific gravity 0.8667 will dissolve approximately 0.95 c. c. of acetylene; that is, an average of 1.10 c. c. of acetylene per 1 c. c. of oil. This absorption is not a constant but varies with different oils. This variation will introduce an error of approximately 110 c. c. per 100 c. c. of oil used; or 17.24 grams of a sample will absorb 18.96 c. c. of acetylene, introducing an error of approximately 18.96 in 580 c. c.; that is, 3.2 per cent in the water content. For this error approximate correction can, of course, be made. If the sample is too viscous to mix readily with the carbide it may be diluted with 50 c. c. of an oil of low vapor tension such as kerosene, but correction must be made for the acetylene absorbed therein. For example, 55 c. c. of acetylene absorbed in 50 c. c. of the diluent plus 18.96 c. c. absorbed in the 17.24 c. c. of a sample will give an approximate correction of 74 c. c., which is to be added to the burette reading. It must also be noted that a portion of the water is held by the calcium hydroxide formed in the action of the water on the carbide.

This method is easily carried out, is approximately correct, and may be used for many classes of materials containing water.

5. TREATING WITH SODIUM.

Graefe [a] estimates the water content of an oil by agitating with finely cut metallic sodium. This process may be carried out as follows:

For each gram of water in the sample of oil, about 2 grams of finely cut sodium is used in the same apparatus and in exactly the same manner as described in method 4. The reaction will be as follows:

$$Na_2+2H_2O=2NaOH+H_2$$

or 1 gram of water will generate $\frac{2}{36}$ gram of hydrogen.

One thousand cubic centimeters of hydrogen weighs 0.09004 gram [b] at 0° C. and 760 mm. pressure, or 1 gram of hydrogen has a volume of 11,106.17 c. c. One gram of water generates $\frac{2}{36}$ (11,106.17) c. c., or 617.0095 c. c. of hydrogen; or 1 c. c. of hydrogen represents 0.00162 gram of water in the sample used.

This method gives much more accurate results than the use of calcium carbide, as hydrogen is not appreciably soluble in petroleum products.

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estimating water in petroleum: Jour. Soc. Chem. Ind., vol. 29, 1910, pp. 197–198; Chem. Abs., vol. 4, June 20, 1910, p. 1664; Petroleum, vol. 5, July 6, 1910, p. 1173; vol. 6, Sept. 6, 1911, p. 2245; Matirières Grasses, 1911, p. 4; Met. and Chem. Eng., vol. 8, May, 1910, p. 283.

[a] Graefe, E., Zur Wasserbestimmung in Rohpetroleum: Petroleum, vol. 1, 1906, pp. 813–817; Jour. Soc. Chem. Ind., vol. 25, Nov. 15, 1906, p. 1035.

[b] Landolt and Börnstein: Physikalisch-chemische Tabellen, 1905, p. 223.

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6. USE OF A COLOR-COMPARATOR TUBE.

Martin [a] compares the sample under consideration in a color-comparator tube with standard mixtures of oil and water. This method of determination of water content is roughly approximate and applicable only when the water is present in visible suspension.

7. TREATING WITH NORMAL ACIDS.

Nettel [b] mixes 100 c. c. of the sample with 100 c. c. of petroleum spirit, adds 50 c. c. of decinormal hydrochloric acid, agitates five minutes, allows the mixture to settle at 65° C. for half an hour, and titrates an aliquot part of the separated acid-water layer with normal alkalies. He concludes that the loss in acidity of the standard solution is due to the absorption of acid by the water dissolved in the oil, basing his conclusions on the assumption, first, that water in petroleum is neither acid nor alkaline; second, that the added acid solution mixes thoroughly with the water in the oil; and third, that crude oil itself has no action on the acid.

Crude oil often contains natural mineral waters which may be either acid or alkaline, and refined oils may often contain some of the wash alkalies. The presence of such mineral waters or wash alkalies would prevent accurate results from the use of the method described above.

8. ELECTRICAL TREATMENT.

A convenient method to reduce the water content of an emulsified oil to approximately 0.5 of 1 per cent has been developed by Cottrell and Speed, [c] Cottrell and Wright, [d] and Cottrell, [e] and is described by Beazley. [f] The method is admirably adapted for reducing the water content on a large scale. A convenient laboratory apparatus may be made as follows:

A glass tube about 5 cm. in diameter and 30 cm. long, and open at the top, is drawn out at its lower end and fitted with a glass cock. On the inner surface of the tube is fitted a copper-gauze cylinder (*c*, fig. 2) extending from the top to within 5 cm. of the bottom.

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[a] Martin, W. H., Estimation of the quantity of oil contained in feed water: British Patent 11067, Dec. 6, 1905.

[b] Nettel, R., Determination of water in petroleum: Chem. Zeit., vol. 28, 1904, p. 867; Jour. Soc. Chem. Ind., vol. 23, 1904, p. 952.

[c] Cottrell, F. G., and Speed, J. B., Separating particles of one liquid, as water, suspended in another: U. S. Patent 987114, Mar. 21, 1911; Chem. Abs., vol. 5, June 10, 1911, p. 1993; Separating mixed liquids: U. S. Patent 987115, Mar. 21, 1911; Chem. Abs., vol. 5, June 10, 1911, p. 1993; Apparatus for treating mixed liquids electrically to effect their separation: U. S. Patent 987116, March 21, 1911: Chem. Abs., vol. 5, June 10, 1911, p. 1993.

[d] Cottrell, F. G., and Wright, A, C., Separating mixed liquids by electric treatment: U. S. Patent 987117, Mar. 21, 1911; Chem. Abs., vol. 5, June 10, 1911, p. 1993.

[e] Cottrell, F. G., Separating one liquid from suspension in another: U. S. Patent 994377, June 6, 1911: Chem. Abs., vol. 5, Aug. 10, 1911, p. 2534.

[f] Beazley, A., Removal of water from crude petroleum: Oil Age, vol. 3, Apr. 21, 1911, pp. 2–4; Petroleum, vol. 6, 1911, p. 2075; Chem. Abs., vol. 5, 1911, p. 3727.

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Between the gauze and the tube is placed a single wrapping of wet cheesecloth or very thin muslin. The tube, held in a vertical position, has centered within it a heavy copper wire or rod, *r*, which also extends from the top to within 5 cm. of the bottom. The copper wire and the copper-gauze cylinder are connected to the high-potential (2,200-volt) terminals of a step-up transformer, *t*.

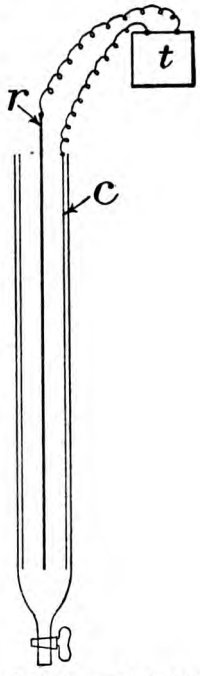

FIGURE 2.—Apparatus for the determination of the water in petroleum by electrical treatment.

In use the tube is filled with the emulsified oil to be examined, and the circuit is closed. The high-tension current breaks down the emulsion and allows the water to trickle down the cheesecloth and settle into the lower end of the tube where it can be drawn off. With oils containing considerable water, the current must be broken at short intervals to prevent overheating of the oil.

This process will readily reduce the water content of an emulsion, with little heating, to as low as 0.5 of 1 per cent in half an hour, and is admirably adapted to reducing the water content of emulsions when heating is objectionable, but quantitatively it is far less accurate than methods 2, 3, 4, 5, 9,and 10.

9. DISTILLING WITH A NONMISCIBLE LIQUID.

Dean[a] estimates the moisture in creosote as a result of distillation with a water-saturated xylene and of measurement of the water that separates in the distillate. Holde[b] and Marcusson[c] use the same method for water in greases, whereas Marcusson[d] uses toluene, as well as xylene.

Testori[e] employs turpentine in a like manner for the determination of the water content of molasses; Prutzman[f] uses a mixture of benzene and toluene for this purpose. Sadtler[g] recommends the use of benzene boiling at a temperature of 300° to 450° F.

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[a] Dean, A. L., Estimation of moisture in creosoted wood: Forest Service Cir. No. 134, 1908; Jour. Ind. and Eng. Chem., vol. 2, February, 1910, p. 66.

[b] Holde, D., Wasserbestimmung in Fetten: Petroleum, vol. 4, 1908, p. 14; Zeitschr. angew. Chem., vol. 31, 1908, pp. 2138–2144; Chem. Abs., vol. 3, 1909, p. 123; Jour. Ind. and Eng. Chem., vol. 1, June, 1909, p. 356.

[c] Marcusson, J., Über die Wasserbestimmung in Ölen, Fetten, und Seifen durch Destillation. Mitt. K. Material-Prüfungsamt, vol. 23, 1905, p. 58; Jour. Ind. and Eng. Chem., vol. 2, February, 1910, p. 66.

[d] Marcusson, J., Die Bestimmung des Wasser- und Saüregehaltes von Schmierfetten: Zeitschr. angew. Chem., vol. 18, 1905, p. 754.

[e] Testori, G., Determination of water in tars by distillation: Staz. sperim. agara. Ital., vol. 37, pp. 366–369; Chem. Centralbl., vol. 75, 1904, pp. 562–563; Jour. Ind. and Eng. Chem., vol. 2, February, 1910, p. 66.

[f] Prutzman, P. W., Distillation test for water in petroleum: Cal. Derrick, vol. 3, February, 1911, p. 5, Petroleum, vol. 6, April 5, 1911, p. 722.

[g] Sadtler, S. S., The determination of moisture by distillation: Jour. Ind. and Eng. Chem., vol. 2, February, 1910, pp. 66–67; Chem. Abs., vol. 4, April 20, 1910, p. 1006.

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A convenient method is as follows:

One hundred grams of the sample is mixed in a distilling flask with 200 grams of toluene (or benzene[a] or xylene) and distilled into a graduate in the usual manner until the distillate comes over perfectly clear. The distillate is allowed to settle by gravity, or is separated by a centrifuge, and the volume of water is read off directly, or the water may be withdrawn with a micropipette and weighed. As water is somewhat soluble in all of the three hydrocarbons mentioned, the method is not accurate but is convenient when only approximate results are desired.

10. DISTILLING OFF THE WATER DIRECTLY.

Bell,[b] Senger,[c] and Allen[d] distill the sample directly and measure or weigh the quantity of water that settles in the distillate.

The water content may be accurately and conveniently determined during the course of an ordinary distillation in the following manner:

Two hundred grams of the sample is weighed into a one-fourth-liter distilling flask and the distillation carried out in the ordinary manner at the rate of one drop of distillate per second. The distillation can be performed most accurately in an electric still.[e] At temperatures between 90° and 150° C. the water distills over and can be removed from the receivers by means of a micropipette and weighed. Usually a few drops of water adhere to the condenser and fail to run into the receivers; in this event a small pellet of absorbent cotton, moistened with water, squeezed as dry as possible, and weighed, is fastened to a wire and run up into the condenser to remove these last traces of water. The amount of the increased weight of the cotton pellet, reckoned as water, is added to the weight of the water in the receivers.

With an oil containing considerable water, it is advisable to cause a slow current of a dry, inert gas, such as carbonic acid, to bubble through the oil in the distilling flask to carry off the vapors of oil and water as soon as formed. The gas current will reduce bumping and the overheating of the oil to a minimum. The condenser must also be kept well cooled throughout the distillation. This method, with the quantity of sample and the apparatus above mentioned, is accurate to less than 0.03 of 1 per cent.

---

[a] Groschuff, E., The solubility of water in benzene, petroleum, and paraffin oil: Chem. Abs., vol. 5; August 10, 1911, p. 2550.

[b] Bell, J. C., Estimation of water in paraffin residues and crude paraffin: Chem. News, vol. 30, 1874, pp. 57–58.

[c] Senger, E., Bestimmung des Wassergehaltes im Teer durch Destillation: Jour. Gasbel., vol. 45, 1902, p. 841; Jour. Soc. Chem. Ind., vol. 21, 1902, p. 1475; Jour. Ind. and Eng. Chem., vol. 2, February, 1910, p. 66.

[d] Allen, I. C., Jacobs, W. A., and Burrell, G. A., Physical and chemical properties of the petroleums of the San Joaquin Valley, with a chapter on analyses of natural gas from the oil fields of southern California: Bulletin 19, Bureau of Mines, 1911, p. 8.

[e] Allen, I. C., Op. cit., p. 28.

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CONCLUSIONS.

Method 1 is approximate and applicable to heavy oils and greases only.

Method 2 is approximate and applicable to thin oils. A diluent is to be avoided.

Method 3 is like method 2, but is more rapid, and somewhat more accurate.

Method 4 is convenient and, with petroleums, accurate to approximately 3 per cent of the water percentage.

Method 5 is convenient and accurate.

Methods 6 and 7 are approximate only.

Method 8 is successful in breaking up an emulsion on a commercial scale or in reducing the water content of an oil to such a condition that it can be successfully treated in some other manner.

Method 9 is accurate to approximately 0.033 gram of water per 100 c. c. of benzene and oil in the distillate.

Method 10 is convenient and can be used simultaneously with a distillation. It is accurate to about 0.003 gram of water in the distillate if the water is cooled to about 2° C.

PUBLICATIONS ON FUEL TESTING.

The following Bureau of Mines publications may be obtained free by applying to the Director, Bureau of Mines, Washington, D. C.

BULLETIN 1. The volatile matter of coal, by H. C. Porter and F. K. Ovitz. 1910. 56 pp., 1 pl.

BULLETIN 2. North Dakota lignite as a fuel for power-plant boilers, by D. T. Randall and Henry Kreisinger. 1910. 42 pp., 1 pl.

BULLETIN 3. The coke industry of the United States as related to the foundry, by Richard Moldenke. 1910. 32 pp.

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